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Preparation and Characterization of Dabco (1,4-Diazabicyclo [2.2.2]octane) modified bentonite: Application for Congo red removal

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Abstract. Natural bentonite provided from Sarolangun deposit was modified with 1,4-Diazabicyclo[2.2.2]octane (Dabco) to form a new class of porous material. Prior further modification, the natural bentonite was cleaned up and activated by NaCl to remove the impurities and increase the bentonite nature. Dabco modified bentonite (Dabco-bent) was prepared by exchanging the inorganic cation placed in the interlayer space of the montmorillonite mineral structure with the 0.01 M Dabco¹⁺ at pH 6. The modified bentonite products were characterized using X-Ray powder diffraction and FT-IR to monitor the change of the bentonite crystallinity and function group due to the modification process. The XRD result confirmed that during the modification process, the $d(001)$ of smectite peak at 2θ around 6° was shifted. After the modification, the $d(001)$ reflection of the montmorillonite interlayer was shifted 0.36° to the left indicating that the interlayer space of the montmorillonite has been expanded during the modification process. The FTIR spectra of Dabco modified bentonite exhibit no significantly different with the host bentonite. However, the presence of the new band at the wavenumber around 3000 and 2800 cm^{-1} indicates that the Dabco molecule has been successfully inserted to the bentonite molecule. The Congo red adsorption experiment was performed onto Dabco-bent product by batch technique. The experiment data described that kinetic model for Congo red adsorption onto Dabco-bent was adequately followed the second-order kinetic model and well described by Freundlich adsorption isotherm model.

Keywords: Natural bentonite, Dabco modified bentonite, Adsorption, Congo red

1. Introduction

Bentonite, as one of the abundant natural clays, has broad application in industry sector for more than 40 years. The technology development has forced the bentonite utilization from the traditional toward higher value product like a catalyst, adsorbent, nanomaterials, medicine, and fine chemicals [1]. Nowadays, in many countries bentonite also used as raw material in cosmetic production due to its moisturizing, detoxification, decontamination, and itching relieving properties. The high demand of the bentonite clay in the various particular area such as science and technology lies in their high adsorption capacity, fire retardant, and naturally abundant and low-cost material [2].

Based on the previous literature and many published papers, the term of bentonite was used to describe the clay material that dominantly composed of smectite mineral wherever its origin [3]. The common bentonite used in industry was dominated by montmorillonite mineral, including calcium bentonite, and sodium bentonite. Montmorillonite mineral that composes the bentonite clay is a 2:1 layers class of smectite. It was constructed by silica tetrahedral sheets (T), and one central alumina octahedral sheet (O) [4]. The surface of bentonite has a permanent negative charge due to the isomorphous substitution of Al^{3+} with Fe^{2+} and Mg^{2+} in the octahedral sheet and Si^{4+} with Al^{3+} in the



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tetrahedral sheet. Naturally, the net negative charge on the bentonite surface was balanced by exchangeable inorganic cations located in the interlayers site [5].

Naturally, bentonite formed by the alteration of the volcanic ash. It consists dominantly by smectite minerals like montmorillonite and some others like saponite, nontronite, and hectorite [6]. Bentonite has hydrophilic properties on its surface. This property causing some restriction in its applications such as reduce the dispersion properties in the organic matrix and make it the as weak adsorbent for removal of organic compounds [6]. Although Bentonite is known as a high utilization natural material with comparatively low-cost than other materials, its restriction in the organic field will reduce the value of the bentonite material in the market. Till now, many methods have been developed in the way of bentonite modification to break the limitation of bentonite utilization.

The common modifications of bentonite material were physical activation, chemical activation, cation exchange, intercalation, plasma surface modification, and pillarization [7–10]. Based on these modification methods, pillarization was one of the most used method due to the high effectivity in changing the surface and interface physicochemical properties of the bentonite. Pillared clay has 20-fold higher surface area than the parent natural bentonite material [11]. In the pillaring process, the inorganic cation lied in the interlayer space of the bentonite was changed by another cation which has the better characteristic. Many kinds of cation have been studied to be used as a pillaring agent for the bentonite including organic cation, inorganic cation, organometallic cation, metallic cluster cation, polyoxide cation, and mix oxide cation [12].

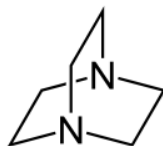


Figure 1. Chemical structure of 1,4-diazabicyclo[2.2.2]octane (Dabco) compound used as bentonite modifier

In this study, a locally grounded bentonite from Sarolangun was modified by pillaring method using an organic based pillaring agent, i.e., 1,4-Diazabicyclo[2.2.2]octane (Dabco) (Figure 1). In order to investigate and to control the degree of modification process, X-Ray diffraction (XRD) and IR spectroscopy (FTIR) were used. The Dabco modified bentonite product then applied as an adsorbent to remove the Congo red dye from aqueous solution. Congo red dye is a well know colorant used in many textile industries and reported as one of the organic molecules that causing an environmental problem when contacted with the water body [13]. The result of this work is hoped to be a reference for the development of local Indonesian bentonite that has no significant economic cost in the market.

2. Materials and Methods

2.1. Materials

Bentonite as the host modified material used in this work was purchased from local bentonite mining located in Sarolangun district, Jambi Province, Indonesia. The natural bentonite pretreatment was conducted to reduce and remove the organic and inorganic impurities. 1,4-Diazabicyclo[2.2.2]octane (Dabco) was obtained from Kanto Chemical Company, Japan. It was used without further purification. Sodium hydroxide (NaOH) and hydrochloric acid (HCl) were purchased from Merck Millipore without further purification. Congo red dye used was in analysis grade provided by Sigma Aldrich and used as received.

2.2. Instrumentation

Chemical analysis of the natural, Na-exchanged bentonite, and modified bentonite were conducted using X-Ray diffraction on Rigaku Miniflex 600 instrument equipped with Ni-filtered CuK α radiation. The

XRD pattern was recorded at 40 kV and 40 mA with fix slit in 2θ range about $5 - 50^\circ$ under scanning speed of $5^\circ/\text{min}$. The d_{001} value of the samples was measured using the Bragg's Law as follow:

$$d = \frac{n\lambda}{2\sin\theta} \quad (1)$$

Where n is the diffraction order, λ is the wavelength of the wavelength of the X-ray, θ is the diffraction angle, and d is the basal spacing [14]. The functional group of natural bentonite, Na-exchanged bentonite, and modified bentonite samples were characterized using FT-IR Shimadzu Prestige-21 instrument using KBr disc in the wavenumber $400-4000 \text{ cm}^{-1}$. The samples were contacted and mixed with the KBr powder and grinded finely in a porcelain mortar. The mixture then mounted to the FT-IR instrument, and the sample measurement started in the percent transmittance mode (%T). The Congo red concentration after contacted with Dabco-bent was measured using spectrophotometer UV-Vis EMCLab.

2.3. Natural Bentonite Preparation

Prior used, the raw bentonite was purified and cleaned by the following procedure. Approximately 500 g of raw bentonite was milled, and the fine-grained raw bentonite powder was collected and sieved through 100 mesh ASTM standard sieve. The fine-grained of raw bentonite then washed using distilled water for several times and dried in hot air oven for overnight. The clean bentonite sample then stores in an isolated flask for further use. The natural bentonite characterization was conducted using XRD and FTIR to study its chemical structure.

To optimize the modification process, the bentonite sample which is calcium based bentonite (Ca-Bentonite) was converted to the Na-bentonite by treating with NaCl. Approximately 100 g of clean bentonite sample was contacted with 500 mL of 1 M NaCl in 1000 mL beaker glass. The mixture then stirred for 24 hours with 200 rpm rotation speed at 60°C . After stirring, the suspension washed using distilled water in a Buchner funnel equipped with a vacuum pump. The bentonite residue then rinsed several times using distilled water to remove the excess of NaCl and Cl⁻ ions. Light yellow bentonite was obtained than dried in the laboratory oven and grained with porcelain mortar. The change of bentonite structure during the Na-exchange process was monitored with XRD and FT-IR instrument.

2.4. Dabco modified bentonite preparation

Dabco modified bentonite was prepared based on the three steps procedure including preparation Dabco¹⁺ solution, Dabco insertion, and drying. Dabco¹⁺ solution (0.01 M) was prepared by diluting appropriate amounts of Dabco powder with distilled water at pH 6 ($K_1 = 10^{8.19}$) [15]. The insertion process was conducted in 500 mL beaker flask in a closed condition. Five grams of Na-bentonite and 250 mL of Dabco¹⁺ solution was mixed under vigorous stirring for 24 hours at room temperature condition. After stirring, the mixture then separated using filtration paper. The treated bentonite residue then washed with distilled water for several times and dried at room temperature. The dry intercalated bentonite then calcinated in a muffle furnace at 150°C [6]. The Dabco modified bentonite product, assigned as Dabco-bent, then characterized using XDR and FT-IR to study the change of the crystallinity and the structure of the bentonite during the pillarization process.

2.5. Congo red adsorption study

The capability of the Dabco modified bentonite on Congo red removal was investigated in batch adsorption technique. The Congo red stock solution was prepared by dissolving 1 g of Congo red powder into 1000 mL of distilled water. The Congo red standard and working solution were made by diluting the Congo red stock solution into desired concentration with distilled water. As much as 0.1 g of Dabco-bent powder was contacted with 50 mL of Congo red solution under vigorous stirring with predetermined contact time in room temperature condition. After finished, the mixture then separated

by centrifugation then the remaining concentration of the Congo red solution was measured using UV-Vis spectrophotometer at wavelength 500 nm.

The Congo red removal percentage and the amount of equilibrium adsorption were calculated based on the following equations:

$$\% \text{Removal} = \frac{(C_i - C_t)}{C_i} \times 100 \quad (2)$$

$$q_t = \frac{(C_i - C_t)}{m} V \quad (3)$$

Where C_i is the initial concentration of the Congo red solution (mg L^{-1}), C_t is the remaining concentration of the Congo red after the adsorption process at any time (mg L^{-1}), q_t is the amount of the Congo red adsorbed on the adsorbent (mg g^{-1}), m is the mass of the adsorbent used (g), and V is the volume of the Congo red solution used (L).

13 Results and Discussion

3.1. Modified Bentonite Characterization

3.1.1. XRD Analysis

The primary technique that commonly used to study the mineralogical composition of the clay minerals is X-ray diffraction (XRD) powder. In this study, the structure of natural bentonite grounded from Sarolangun District was studied with XRD. The XRD powder pattern of the natural bentonite sample was depicted in Figure 2. The XRD pattern confirms that the typical characteristic peak of the montmorillonite (001) observed at the 2θ value around 6° and 27° [16]. As reported by Djukic et al. [17], the other peak observed at 2θ around 19° , 20° , and 26° were the associated mineral of bentonite, i.e., montmorillonite, calcite, dolomite, and quartz.

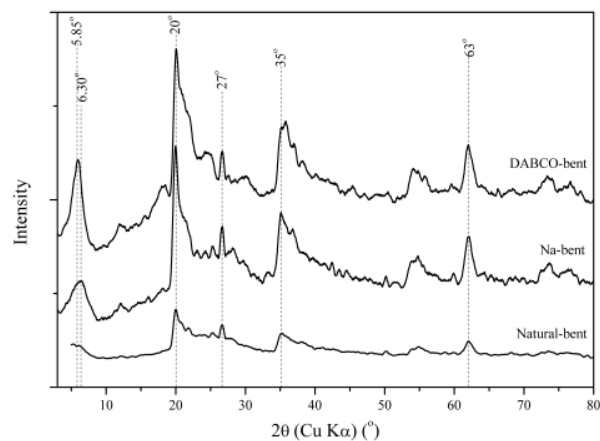


Figure 2. XRD powder pattern of Na-exchanged bentonite

The change of bentonite structure during the Na-exchange process was studied using XRD powder. The XRD powder pattern of the Na-bentonite described in Figure 2. The XRD pattern of the Na-bentonite exhibit the characteristic peak $d(001)$ of the montmorillonite at 2θ value of 6.30° . Based on the value of the natural bentonite (Figure 2), the 2θ value of the Na-bentonite was shifted. This shifting also observed on the work done by Hao et al. [18] and this phenomenon is due to the exchanged of the

interlayer cations (Ca^+) of the natural bentonite with Na^+ that has smaller cation size ($\text{Na}^+ = 7.16 \text{ \AA} < \text{Ca}^{2+} = 8.24 \text{ \AA}$). This exchange process inflicts the constriction of the interlayer space [19].

As illustrated in Figure 2, the X-ray diffraction patterns of Na-bentonite and Dabco modified bentonite has the same typical diffraction peaks of bentonite at 2θ about 6° [18]. The peak of this pattern was shifted significantly by the Dabco intercalation. The Na-bentonite as the host material has 2θ value of 6.3° and shifted to 5.85° after modified by Dabco with concentration 0.01 M. The reduce of the 2θ value of the typical montmorillonite peak represent that the basal spacing of bentonite interlayer space increased. This result indicated that the Dabco molecule has intercalated into the interlayer space of the bentonite by replacing the Na^+ cation that constructed the host bentonite [20].

3.1.2. FTIR Analysis

One of the most powerful chemical analysis is Fourier transform infrared spectroscopy (FTIR). It is the most useful technique which is used to gain an infrared absorption spectrum of a material (liquid, solid, or gas). FTIR work by bombarding the IR radiation through a sample and some radiation passes through or transmitted, and some others are absorbed. Since it founded, the utilization of IR spectroscopy has been raised due to each chemical molecule produce different spectral fingerprints. In this study, the IR spectroscopy was used to investigate the spectral fingerprint of a molecule that constructed the bentonite sample.

The FTIR spectra of the natural bentonite from Sarolangun District was shown Figure 3. The natural bentonite spectrum exhibit an adsorption bands at 3448 and 1635 cm^{-1} . These bands were assigned to the stretching and bending respectively of the OH group from the water molecule that contained in the interlayer space of the bentonite. A small bit band at 3626 cm^{-1} interpreted the stretching vibration of the -OH group that linked to the Al^{3+} in octahedral sheets. The strongest band was recorded at the wavenumber of 1033 cm^{-1} . This band could be easily being assigned as the Si-O stretching plane band that commonly recognized in the infrared spectrum around 1000 cm^{-1} . The relatively weak band at 532 and 470 cm^{-1} corresponded to the Si-O-Al in the octahedral sheet and Si-O-Si bending vibration, respectively [21].

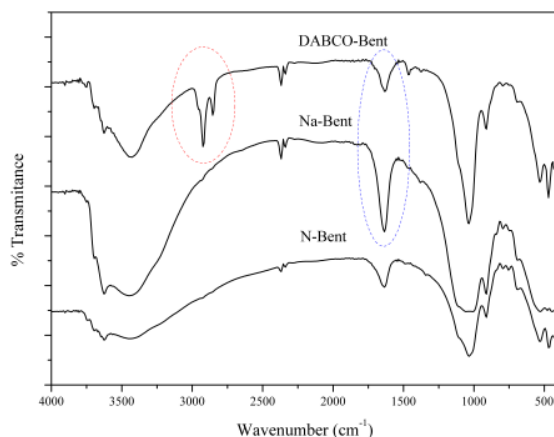


Figure 3. FT-IR spectra of natural bentonite (N-Bent), Na-exchanged bentonite (Na-Bent) and DABCO modified bentonite (DABCO-Bent)

The FTIR spectra of NaCl-treated natural bentonite shown in Figure 3. The figure showed that the characteristic band of the natural bentonite was not shifted. However, the intensity of the characteristic band at the wavenumber 3448 and 1635 and 1033 cm^{-1} as the characteristic peak of the water molecule and silica was significantly increased. This phenomenon described that during treated with NaCl, the

water molecule content in the bentonite structure was increased. Moreover, at the wavenumber around 524 cm^{-1} , the band intensity of the Na-bent was reduced. This phenomenon indicated that the Si-O-Al group was reduced by the presence of the Na^+ cation in the interlayer space of the bentonite structure [22].

After the modification process, as illustrated in Figure 3, the FTIR spectra of the Dabco-bent was slightly different with the Na-bent as the host. The main characteristic band of the Na-bent was not shifted significantly, but a new band at wavenumber around 3000 and 2800 cm^{-1} was recorded at the spectra of Dabco-bent. These band was the characteristic band of the C-H bending vibration. This finding describes that the Dabco molecule has successfully intercalated into the bentonite interlayer structure. Moreover, compared with the Na-bent spectra, Dabco-bent spectra has less intensity almost in all the recorded band. Particularly in the characteristic of the OH molecule. This finding indicated the removal of the water molecule in the calcination treatment during the modification process.

3.2. Adsorption Kinetics Study

The dependency of Congo red adsorption toward the contact time was investigated by conducting the adsorption experiment in various predetermined time, i.e., 0 to 60 minutes. In addition, this investigation is useful to calculate the kinetics of adsorption and the maximum adsorption capacity of the Dabco-bent for Congo red removal. The adsorption kinetics was investigated by calculating the constant of the adsorption rate and the adsorption capacity at equilibrium state. The experiment data was obtained by contacting 0.1 g of Dabco-bent with 50 mL of Congo red (50 mg L^{-1}) at room temperature with different contact time ranging from 0 – 60 minutes.

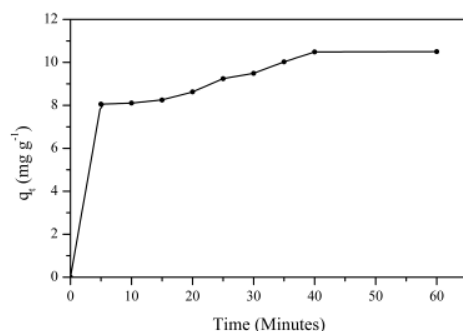


Figure 4. Effect of contact time on the adsorption capacity Dabco-bent

The result was presented as a graph of the correlation between the contact time to the adsorption capacity as depicted in Figure 4. The figure showed that the adsorption of Congo red on Dabco-bent was rapidly occurred in the beginning (around the first 15 minutes), then getting slowed down until reached the equilibrium after 60 minutes with maximum adsorption capacity 10.5 mg g^{-1} . As reported by Wang et al. [23], this phenomenon occurred due to in the begging of the adsorption process, the gradient concentration of the Congo red solution is larger. As the time went, the concentration gradient was reduced and the adsorption rate was gradually decreased until establishing the sorption equilibrium at 60 minutes. Therefore, the 60 minutes contact time was used for subsequent experiments.

Further, the obtained data of the contact time effect was evaluated to determine the kinetic adsorption parameters based on the adsorption kinetic models that have been established, i.e., the pseudo-first-order kinetic model, second-order kinetic model, and intraparticle diffusion model. The pseudo-first-order kinetic model is the first model that used to describe the adsorption kinetic model in a liquid/solid system. This model is mostly used in the aqueous solution adsorption system with weak concentration. The linear form of the pseudo-first-order kinetic model proposed by Lagergren [24] is presented as follows:

$$\log(q_e - q_t) = \log q_e - K_1 \frac{t}{2.303} \quad (4)$$

Where q_e is the amount of Congo red adsorbed onto Dabco-bent (mg g^{-1}) at equilibrium, q_t is the amount of the Congo red adsorbed (mg g^{-1}) at the respective time, and K_1 is the rate constant of the pseudo-first-order kinetic model. The value of the K_1 and predicted q_e based on the pseudo-first-order kinetic model could be determined based on the slope and intercept value from the plotted $\log(q_e - q_t)$ versus t .

The pseudo-second-order kinetic model is an adsorption kinetic model that refers to the capacity of the adsorption equilibrium. This model is express as follow:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (5)$$

Where K_2 is the constant of the adsorption rate based on the pseudo-second-order model (g/mg min^{-1}). By plotting t/q_t against t , the adsorption rate constant (K_1), and the predicted q_e can be calculated based on the intercept and slope obtained.

The mathematical equation for intra-particle diffusion is developed by Weber and Morris [25] as follow:

$$q_t = K_{id} t^{1/2} + C \quad (6)$$

Where K_{id} and C are the intraparticle diffusion rate constant ($\text{mg/g min}^{1/2}$) and a constant refers to the boundary thickness of the layer. If the plot of q_t against $t^{1/2}$ is a linear plot and passes through the origin, it can be described that the adsorption mechanism is only controlled by the intraparticle diffusion.

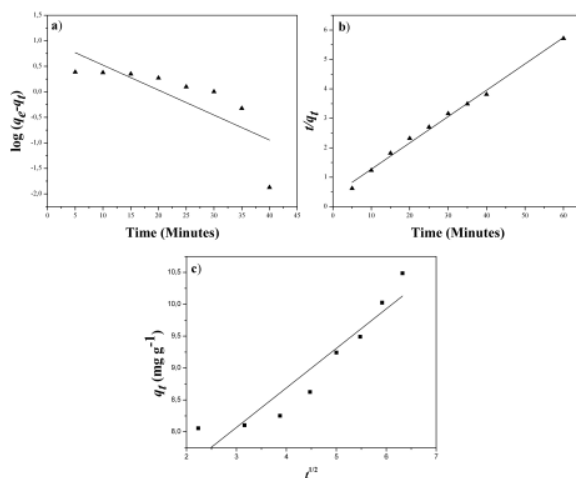


Figure 5. Fit of the experimental data to the pseudo-first-order (a), pseudo-second-order (b), and intraparticle diffusion (c) model

The plot and kinetic adsorption parameters based on the three kinetic models used are presented in Figure 5 and Table 1, respectively. The obtained data showed that the kinetic model for Congo red adsorption on Dabco-bent is best fitted by the pseudo-second-order model rather than the pseudo-first-

order model. In this case, the R^2 value for the pseudo-second-order model is the closest to unity (0.9871), greater than the R^2 value for the pseudo-first-order model (0.8554). For the intraparticle diffusion model, the result showed that the C value is higher enough to declare the proportion to the boundary layer thickness. Moreover, the linear plot of the intraparticle-diffusion model (Figure 5c) was not pass through the origin. It indicates that the adsorption process was not completely controlled by the diffusion mechanism [26].

Table 1. Kinetic parameters for Congo red removal on Dabco-bent

Pseudo-first order		Pseudo-second order		Intraparticle diffusion	
q_e (mg g ⁻¹)	10.3121	q_e (mg g ⁻¹)	11.0358	K_{id}	0.5562
K_1 (min ⁻¹)	0.1126	K_2 (g mg ⁻¹ min ⁻¹)	0.0230	C	6.4659
R^2	0.8554	R^2	0.9871		

3.3. Adsorption isotherm study

The term adsorption isotherm is a way to explore the adsorption process through a graph that gives a correlation between the amount of the adsorbate and pressure when the system was in the equilibrium state with constant temperature. The investigations were done by conducting the adsorption experiment in various initial dye concentration with predetermined the other variable, i.e., contact time, temperature, and amount of the adsorbent. The result of the experiment was presented in Figure 6. As shown in the Figure, by increasing the initial dye concentration, the adsorption capacity was gradually increased. As reported in our previous study, this result was probably affected by the increase of the driving force due to the increasing the number of dye molecules in the solution. In the present work, the adsorption isotherm based on the experiment result was investigated by Langmuir and Freundlich model.

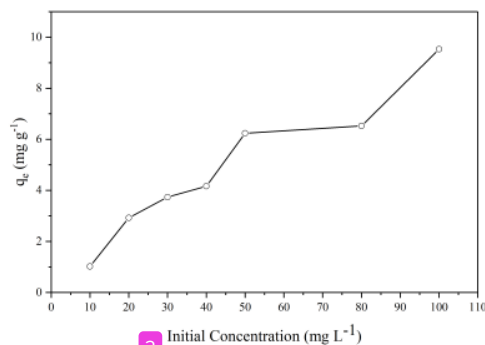


Figure 6. Effect of initial dye concentration

The Langmuir adsorption isotherm model is a model that describes the adsorption process as a monolayer sorption process. It means that the adsorbate adsorbed on the surface of adsorbent takes places in one active site of the adsorbent. The adsorption stops if the active site of the adsorbent has been covered by one adsorbate molecule or ion. The linear form of the Langmuir adsorption isotherm model is presented as follows:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (7)$$

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Where C_e is the concentration of the dye solution at the equilibrium state (mg L^{-1}), C_0 is the concentration of the unadsorbed dye in the solution (mg L^{-1}), and the Langmuir constant (L mg^{-1}). The values of the Langmuir constant and the predicted maximum adsorption capacity could be determined from the intercept and the slope values of the plot C_e/q_e versus C_e .

The Freundlich adsorption isotherm model is the model that used to describe the sorption process on the heterogeneous surface with a multilayer sorption. The adsorption process is assumed to occur on the heterogeneous surface, and the adsorbate molecule or ion is adsorbed on the surface of the adsorbent in the multilayer form [27]. The linear form of the Freundlich model is depicted in the following equation:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (8)$$

Where K_F (mg g^{-1}) is the Freundlich constant, and n is the constant refers to adsorption intensity. The $1/n$ values indicate the type of adsorption based on the favorability, $1/n$ equals to zero means that the adsorption process is irreversible, adsorption process is favorable if the $1/n$ value more than zero and less than one, and unfavorable if the $1/n$ value more than one.

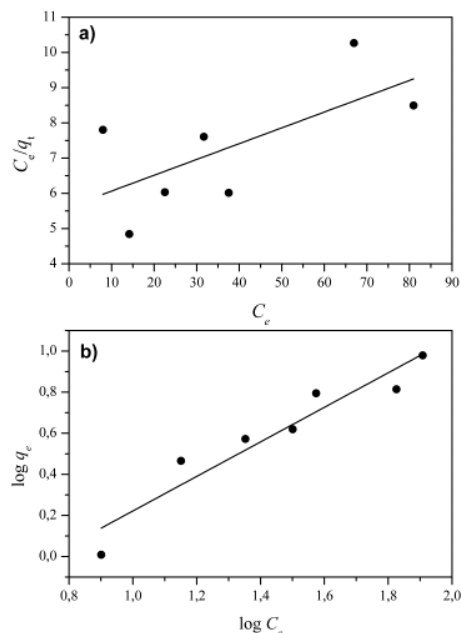


Figure 7. Fit of the experimental data to Langmuir a), and Freundlich b) isotherm model

The adsorption isotherm parameters based on the linear fitting of Langmuir and Freundlich model were summarized in Table 2. It can be seen that the best-fit adsorption isotherm model to describe the adsorption of Congo red on the Dabco-bent was Freundlich model with higher R^2 value than the Langmuir model. This finding is also in agreement with other results on dye adsorption on organo-modified clay [5,28]. This finding means that the adsorption process of Congo red on Dabco-bent was a nonideal adsorption mechanism occurred in the heterogeneous surface with a multilayer sorption form. Based on the linear fit graph of the adsorption isotherm (Figure 7) and the n value of the Freundlich model (Table 2) is higher than one, it can be concluded that the adsorption mechanism of the Congo red on Dabco-bent was dominantly a partition mechanism [5].

Table 2. Adsorption isotherm parameters for Congo red adsorption

Langmuir Isotherm model		Freundlich isotherm model	
q_m (mg g ⁻¹)	22.2816	K_F	0.2404
K_L (L mg ⁻¹)	0.0079	n	1.1893
R^2	0.4491	R^2	0.9118

4. Conclusion

In summary, the characterization of the natural bentonite grounded from Sarolangun District, Na-exchanged bentonite, and Dabco modified bentonite has been conducted using powder X-ray diffraction and FT-IR. The natural bentonite composition based on the XDR pattern result was montmorillonite with some associated mineral such as calcite, dolomite, and quartz. The characterization Dabco modified bentonite using powder XRD show that the peak of montmorillonite was shifted indicate that the Dabco molecule was successfully inserted into the montmorillonite interlayer space. The FTIR analysis of the Dabco modified bentonite represented that the specific band of the natural bentonite was not affected by the pillaring process and during the pillaring process the structure of bentonite host was not changed significantly. The application of Dabco-bent as an adsorbent for Congo red dye removal from wastewater shows that the Dabco-bent has a good adsorption capacity. The further study gives the result that the adsorption kinetic of Congo red on Dabco-bent fit well with the pseudo-second-order kinetic model. The result for adsorption mechanism study based on the adsorption isotherm model revealed that adsorption of Congo red on Dabco-bent follow the Freundlich isotherm model which is mean that the adsorption process was a non-ideal process.

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